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REPORT DOCUMENTATION PAGE				
1a. REPORT SECURITY CLASSIFICATION Unclassified		1b. RESTRICTIVE MARKINGS		
2a. SECURITY CLASSIFICATION AUTHORITY JUN 21 1989		3. DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release; distribution unlimited.		
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE H		5. MONITORING ORGANIZATION REPORT NUMBER(S) ARO 23255-22-MS		
4. PERFORMING ORGANIZATION REPORT NUMBER(S)		5. MONITORING ORGANIZATION REPORT NUMBER(S)		
6a. NAME OF PERFORMING ORGANIZATION Univ. of Cincinnati	6b. OFFICE SYMBOL (If applicable) NA	7a. NAME OF MONITORING ORGANIZATION U. S. Army Research Office		
6c. ADDRESS (City, State, and ZIP Code) Dep't. of Chemistry Cincinnati, OH 45221		7b. ADDRESS (City, State, and ZIP Code) P. O. Box 12211 Research Triangle Park, NC 27709-2211		
8a. NAME OF FUNDING/SPONSORING ORGANIZATION U. S. Army Research Office	8b. OFFICE SYMBOL (If applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER DAAL03-86-K-0032		
8c. ADDRESS (City, State, and ZIP Code) P. O. Box 12211 Research Triangle Park, NC 27709-2211		10. SOURCE OF FUNDING NUMBERS		
		PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.
		WORK UNIT ACCESSION NO.		
11. TITLE (Include Security Classification) Novel Methods for Reinforcing Elastomers				
12. PERSONAL AUTHOR(S) J. E. Mark				
13a. TYPE OF REPORT Final	13b. TIME COVERED FROM 2/10/86 TO 2/9/89	14. DATE OF REPORT (Year, Month, Day) 1989/4/5		15. PAGE COUNT
16. SUPPLEMENTARY NOTATION The view, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy, or decision, unless so designated by other documentation.				
17. COSATI CODES		18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)		
FIELD	GROUP	SUB-GROUP		
			Reinforcement/ Elastomers Fillers	
			Magnetic particles/ Ultimate properties, Toughness. (TE)	
19. ABSTRACT (Continue on reverse if necessary and identify by block number) The primary goal of this project was the development of new techniques for toughening elastomeric materials. The first approach taken was the in-situ precipitation of reinforcing filler particles into the elastomers. Preliminary studies focused on the elastomer poly(dimethylsiloxane) (PDMS) because of the large number of related studies already carried out on this material. Promising techniques were then extended to other elastomers, such as the polyisobutylene. The second approach was the alignment of ferrite-type filler particles to improve reinforcement, at least in chosen directions. This was done during the curing process and utilized a magnetic field of variable strength.				
20. DISTRIBUTION/AVAILABILITY OF ABSTRACT <input type="checkbox"/> UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT. <input type="checkbox"/> DTIC USERS		21. ABSTRACT SECURITY CLASSIFICATION Unclassified		
22a. NAME OF RESPONSIBLE INDIVIDUAL		22b. TELEPHONE (Include Area Code)	22c. OFFICE SYMBOL	

GRANT DAAL03-86-K-0032**J. E. Mark****University of Cincinnati****I. Statement of Problem**

The basic problem investigated in this research was the development of new fillers and filler reinforcement techniques for improving the ultimate properties of elastomers. Of particular interest was the use of ideas from the new sol-gel area of ceramics, which involves the generation of ceramic-type materials from organometallic substances. The utility of the techniques developed was to be gauged by mechanical property measurements on the resulting filled elastomers.

II. Summary of Results

Silica generated by the in-situ hydrolysis of tetraethoxysilane was found to give very good reinforcement in poly(dimethylsiloxane) (14)*, polyisobutylene (1), and poly(methylphenylsiloxane) (5). Similarly produced titania (2,18,20) and alumina (12) also gave good reinforcement, but with some evidence that the particle-elastomer bonding was not as strong as that observed in the case of silica. Zeolites, simply blended into poly(dimethylsiloxane), gave surprisingly good reinforcement (13), and have the advantage of being crystalline with variable channel sizes for penetration by the polymer chains to be subsequently used to form the elastomeric network. The low-temperature behavior of some of the silica-filled materials was also investigated (7). The silica particles were found to interfere with crystallization of the elastomer in the undeformed state.

*Reference numbers correspond to those in Section III.

It was also found to be possible to obtain good reinforcement by the in-situ polymerization of monomers yielding glassy polymers within the elastomeric matrix (11,16,17). Some of these polymerizing chains could also be bonded either to the filler particles (8), or to the cross links (10).

Employing magnetic filler particles was found to be very useful in that the anisotropy of the reinforcement could be controlled by curing the filled polymer in a magnetic field (3,4,6).

The results obtained were also described in a number of review articles (9,15,19,21,22)

III. List of Publications

1. In-Situ Generation of Reinforcement in Polyisobutylene Networks, C.-C. Sun and J. E. Mark, J. Polym. Sci., Polym. Phys. Ed., **25**, 1561 (1987).
2. In-Situ Precipitation of Reinforcing Titania Fillers, S.-B. Wang and J. E. Mark, Polym. Bulletin, **17**, 271 (1987).
3. Anisotropic Reinforcement in Elastomers Containing Magnetic Filler Particles, G. B. Sohoni and J. E. Mark, J. Appl. Polym. Sci., **34**, 2853 (1987).
4. Precipitation of Iron Oxide Filler Particles Into an Elastomer, S. Liu and J. E. Mark, Polym. Bulletin, **18**, 33 (1987).
5. Reinforcement of a Non-Crystallizable Elastomer by the In-Situ Precipitation of Silica, S. J. Clarson and J. E. Mark, Polym. Comm., **28**, 249 (1987).
6. Some Reinforcing Characteristics of Iron-Based Fillers, G. S. Sur and J. E. Mark, Polym. Bulletin, **18**, 369 (1987).
7. The Effect of Network Chain Length Distribution and In-Situ Precipitated Silica on the Low-Temperature Behaviour of Poly(Dimethylsiloxane) Networks, S. J. Clarson, J. E. Mark, and K. Dodgson, Polym. Comm., **29**, 208 (1988).
8. Grafting of Polymer onto Filler Particles Used to Reinforce an Elastomer, G. S. Sur and J. E. Mark, Polym. Bulletin, **20**, 131 (1988).



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9. In-Situ Generation of Ceramic Particles for the Reinforcement of Elastomeric Matrices, J. E. Mark, in "Ultrastructure Processing of Advanced Ceramics", ed. by J. D. MacKenzie and D. R. Ulrich, Wiley, New York, 1988.
10. Grafting of Polystyrene onto the Cross Links in a Siloxane Elastomer, G. S. Sur and J. E. Mark, Eur. Polym. J., **24**, 913 (1988).
11. Elastomer Reinforcement from a Glassy Polymer Polymerized In-Situ, F.-S. Fu and J. E. Mark, J. Polym. Sci., Polym. Phys. Ed., **26**, 2229 (1988).
12. Reinforcement from Alumina-Type Fillers Precipitated Into an Elastomer, J. E. Mark and S.-B. Wang, Polym. Bulletin, **20**, 443 (1988).
13. Zeolites as Reinforcing Fillers in an Elastomer, A. M. S. Al-ghamdi and J. E. Mark, Polym. Bulletin, **20**, 537 (1988).
14. Comparisons Among the Reinforcing Effects Provided by Various Silica-Based Fillers in a Siloxane Elastomer, C.-C. Sun and J. E. Mark, Polymer, **30**, 104 (1989).
15. Recent Studies of Rubberlike Elasticity, J. E. Mark, Pure Appl. Chem., **61**, 000 (1989).
16. Polystyrene-Polyisobutylene Network Composites from In-Situ Polymerizations, F.-S. Fu and J. E. Mark, J. Appl. Polym. Sci., **35**, 000 (1988).
17. Reinforcement of Elastomeric Poly(dimethylsiloxane) by Glassy Poly(diphenylsiloxane), S. Wang and J. E. Mark, J. Mat. Sci., **24**, 000 (1989).
18. Reinforcement of Poly(methylphenylsiloxane) Elastomers by the In-Situ Precipitation of Silica and Titania, S. J. Clarson, D. W. McCarthy, and J. E. Mark, Preprints, Polym. Div., ACS, **30**, 298 (1989).
19. Molecular Theories of Rubberlike Elasticity and Some Recent Results on Model Networks and Unusual Fillers, J. E. Mark, Kautschuk + Gummi.Kunststoffe, **00**, 000 (1988).
20. Reinforcement of Elastomeric Poly(Methylphenylsiloxane) by the In-Situ Precipitation of Titania, S. J. Clarson and J. E. Mark, submitted to Polym. Comm.
21. Overview of Silicon-Containing Polymers, J. E. Mark, in "Advances in Silicon-Based Polymer Science", ed. by J. Zeigler and G. Fearon, American Chemical Society, Washington, 1989.
22. Rubberlike Elasticity, B. Erman and J. E. Mark, Ann. Rev. Phys. Chem., **40**, 000 (1989).

Preprints and reprints of all papers, and six semi-annual reports, have previously been sent to the Army Research Office Library.

IV. List of Participating Scientific Personnel

- A. J. E. Mark, Principal Investigator

- B. S. J. Clarson, Postdoctoral Student
C.-C. Sun, Postdoctoral Student
G. S. Sur, Postdoctoral Student

- C. S.-B. Wang, Visiting Scholar
F.-S. Fu, Visiting Scholar

- D. G. B. Sohoni, Graduate Student, Ph.D. in 1988
A. Davis, Graduate Student
S. Wang, Graduate Student